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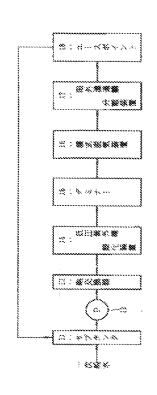
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(51) Int.CL*		識別記号	疗内整理器号	F I			技術技	耐酸所
C02F 1	/44			C03F	1/44		3.	
B01D 19	/00			B01D	19/00		H	
C03F 1	/20			COSF	1/20		Z	
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(22) ALMAH		平成7年(1995)7月17日				enast Frakenta	mmedia wake es	
(men) tressure 6%		THE PART OF CLOSES F.	13 13 LJ	(72)発明者			1814-00-179	
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(54) 【発明の名称】 超純水製造装置

(57) 【聚約】

【課題】 密存御素量が著しく低い超純木を製造する。 【解決手段】 前処理工程より得られた一次純水をサブ タンク11、ボンブ12、熱交換器13、低圧紫外線酸 化装置14 イオン交換装置15、膜式脱気装置16及 び製外濾過膜分離装置17に順次に通水し、得られた極 低溶存酸素の超純水をユースポイント18に送る。



3.

「特許請求の範囲)

「請求項1〕 一次純水を導入し、少なくとも紫外線照 射験化装置とイオン交換純水装置とを有するサブシステ ムに通水して超純水を得る超純水製造装置において。 詩イオン交換純水装置の後段に搬送脱気装置を配置した。 ことを特徴とする超純水製造装置。

(発明の詳細な説明)

100011

(発明の属する技術分野) 本発明は超純水製造装置に係 本製造装置に関する。

(0002)

《從来の技術》従来、半導体洗浄用水として用いるれて いる超終水は、図2に示すように前処理システム!、一 次紙水システム2及びサブシステム3から構成される額 純水製造装置で原水(工業用水、市水、井水等)を処理 することにより製造されている。悶2において、各シス テムの役割は次の通りである。

【0003】凝集。加圧浮上(沈殿)、濾過装置等より 下物質の除去を行う。逆漫透膜分離装置、粉気装置が75 イオン交換装置(選床式又は4床5壊式)を備える一次。 練水システム2では原水中のイオンや有機成分の除去を 行う。なお、連接選牒分離装置では、塩類除去のほかに イオン性、コロイド性のTOCを除去する。イオン交換 装置では、塩類除去のほかにイオン交換樹脂によって吸 第又はイオン交換されるTOC成分を除去する。脱氧装 置(窒素脱氧又は真空粮気)では密存物素の除去を行

ャー(非再生式イオン交換樹脂装置)及び個外濾過膜分 無装置を備えるサブシステム4では、水の純度をより… 層高の超純水にする。なお、低圧紫外線酸化装置では、 低圧紫外線ランプより出される185 n m の勢外線によ りTOCを有機酸さらにはCO、まで分解する。分解さ れた有機物及びCO、は後砂のイオン交換樹脂で除去さ れる。原外濾過膜分離装置では、微小粒子が除去されイメ

GUESSASS 198 miles 1981 19 2H. 0 整化蒸煮+H. +2*0H 至幾個應+H. +H. O.

[60]]] 即り、低圧紫外線酸化装置入口では水(日 。0)とTOCか存在するが、一次純水中のTOCが榧 めて低い場合。紫外線像化装置における紫外線照射響が 設計値よりも過剰となる。(例えば、紫外線照射量を下 ○○↓○ppbに対応した照射量とした紫外線酸化装置 にTOC5ppbの一次純水が流入する場合。 糖外線照 射難がTOC5ppb分だけ過剰となる。)そして、丹 , ○か、この機制な能外線の限制により○日ラジカルを 介してH。O。(滋鬱化水素)となり、溶存職業はTO ○分解に使用されるために見掛け上落存骸素濃度が低下。50

* オン交換樹脂の流出粒子も除去される。

[00051

(発明が解決しようとする課題) 超純水中の密存散素 は、シリコンウェハーの自然酸化酸の摩ぎをコントロー ルする上で重要な因子であるか。上記従来の超純水製造 装置によれば、溶存酸素濃度から~10ppb程度とな ってしまい。さらに低い溶存酸素額が要求されるケース では要求水質を満足できなくなる。

【0006】本発明は、超純水中の溶存酸素を効率的に り、特に溶存酸率適度がきわめて低い超純水を得る超純 30 除去できる超純水製造装置を提供することを目的とす 8.

100071

【課題を解決するための手段】本美明の超純水製造装置 は、一次純水を導入し、少なくとも紫外線照射微化紫癜 (勢外線酸化装置)とイオン交換補水装置とを有するサ プシステムに選木して超純水を得る超純水製造装置にお いて、該イオン交換減水装置の後段に膜式脱気装置を配 置したことを特徴とする。

【0008】本発明者らは、閏2に示されるような超純 なる前処理システム1では、原水中の**継機物質や**コロイー20一水製造装<mark>圏における熔存</mark>酸素濃度の推移を調べた結果。 サブシステム内の無圧架外線酸化装置出口で一旦溶存置 素濃度は低下するが、後段のボリッシャー出口で再度低 狂繁外線酸化装置入口の溶存酸素濃度まで (場合によっ ではそれ以上に)上昇してしまい。結果的に低圧紫外線 部化装置の溶存酸素除去効果が表れないことを確認し た。即ち、一次純水システムの流出水の綿存酸素濃度は 約5~10ppbであり、この水が低圧紫外線酸化装置 で処理されることにより、密存酸素濃度は0~5ppb に低減するが、ボリッシャー出口水の宿存酸素濃度は再 【0004】熱交換器 低圧燃外線酵化装置。ポリッシー30~び5~10ppb程度に上昇しており、結果として得ち れる超純水の密存散素温度は5~10ppbとなる。 【0009】本発明者らは、この現象について鋭意検討 した結果、下記(1)式のような反応が低圧紫外線酸化 装置からボリッシャーの間で生じていることを発出し

[0010]

((t))

100

ボリッシャー内のアニオン交換樹脂) の接触触媒作用で 分解され 西渡〇、が発生するため、ボリッシャー出口 本の総存酸素濃度が上昇する。

する。しかし、住成した日、〇、はボリッシャー(特に

【0012】本発明は、紫外線酸化装置からイオン交換 装置を通ることにより発生した密存物素を膜式脱気装置 で除去することにより、得られる超純水中の溶存物業値 を低下させるようにしたものである。

[0013]

【発明の実施の形態】以下、閉面を参照して本発明を詳

額に説明する。

(9914)図)は本発明の超減水製造装置の一実施例 を示す系統図である。

【0015】各種前処理工程より得られた一次純水(通常の場合、TOC機度2ppb以下の純水)をサブタンク11、ボンブ12 熱交換器13、低圧繁外線酸化装置14、イオン交換装置(本実施例ではアニオン交換樹脂とカチオン交換樹脂とをイオン負荷に応じて混合したデミナー)15、際式脱気装置16及び限外濾過緩分離装置17に順次に選水し、得られた極低溶存酸素の超極本をユースポイント18に送る。

【0018】膜式脱気装置としては、脱気線の一方の側に水を流し、他方の側を真空ポンプで排気し、治存酸素を膜を透過させて真空側に移行させて除去するようにしたものか用いられる。なお、この腰の真空側には若干の水分が脱気膜を透過して出てくるので、この真空側に窒素等のガスを流し、水分を除去して膜性能の低下を防止するのが好ましい。N。流量は一定でも良く、変動させても良い。

【9017】脱気酸は、酸素、窒素、蒸気等のガスは通 20 通するが水は透過しない腹であれば良く、例えば、シリ コンゴム系、ポリテトラフルオロエチレン系、ボリオレ フィン系、ポリウレタン系等がある。この脱気膜として は市販の各種のものを用いることができる。

【0018】この際式膜気装置の真空度は85~70下 の11、N,等のカスの真空側流量は水流量の5~25 %とするのが好ましい。なお、この運転条件は、膜性能 により任意に設定できるが、通常上記のような範囲が好ましい。真空度が過度に低いと脱気効率が低下し、逆に 適度に大きいと膜を通して水も透過側に出てきて効率が 悪くなる。N、流量は適度に少ないと水分除去が十分で なく脱気効率が低下し、大きすぎると真空度が上がらず 脱気効率が低下する。

【10020】本発明においては 紫外線酸化製置で紫外

線照射された水をイオン交換樹脂に接触させた後、膜続 気処理することが重要であり、膜式脱気装置を始外線酸 化装置とイオン交換樹脂との間に配置しても、得られる 超純水中の符存酵素は低減しない。

[0021]

【実施例】以下に比較例及び実施例を挙げて本発明をより具体的に説明する。

[0022]比較例1

デミナー)15、藤式脱気装置15及び限外濾過膜分離 原水(厚木市水:TOC700~800ppb、溶存散 装置17に順次に選水し、得られた極低溶存酸素の超純 10 素8000ppb、電線度240μS/cm)を図2の 水をユースポイント18に送る。 装置によって処理して超純水を製造した。

【0023】実施例1~3

サブシステムを、腹式腕気装置を組み込んだ図1のもの とし、膜式腕気装置の運転条件及び適水量を表1の通り としたほかは比較例1と同様にして超純水を製造した。 【0024】にの比較例及び実施例における各装置の仕

様は下記の通りである。 【0095】何日特殊がWWW 1990 1900 144

【10025】低圧紫外線照射酸化装置:80V×4本 (0、32kW)

50 (発生液長:1850m及び254mm) ボリッシャー(デミナー):カチオン交換樹脂とアニオン交換樹脂とを混合充填した混床式イオン交換装置 SV=70~80hr?

眼外濾過膜分離装置: KU-1010 (栗田工業株式会 社製)

腺気膜: Hoechst Celanese 製Ljqui-Coll 本

- ハウシング:5PCH-120

カートリッジ: 5PCH-118, SN:122) 【0026】

(表)

	水流量	接式脱氧装置			
	(of / fix)	真空度 (Torr)	N s 流激 (Nor*/10)		
美施例:	1.8	70	0.5		
美新例2	4	5.5	0. 6		
実施例3	3	6.5	0.5		
比較例1	3				

【0027】得られた超純木の電導度と低圧業外線酸化 装置以降の各装置の流出水の溶存酸素は表2に示す通り であった。

[0028]

[義2]

) 	***					6
		着 存 機 第 (ppb)				
	(X M -X	第91新版化 英麗出口	70-ttm	### #	UFAC	調制水 電場所 (#8/cm)
実施例(< 5	< 5	9	1		18,24
3.00 (9) 2	< 5	< 8	7	<1	< 3	18-24
英語例3	< 5	< 8	7	<1	< 1	18. 24
£1:8089 1	< 5	≪ 8	7~10		7 1 3	30 83

〔0029〕遊2より、本発明例によると密存骸素騰度 が極めて低い類純水が得られることが明らかである。

[0030]

(発明の効果)以上詳遠した護り、本発明の超純水製造 装置によると、密存酸素濃度が著しく低い超純水を製造 できる。

(短頭の簡単な影響)

(図1)本発明の超純水製造装置の一実施例を示す系統 図である。

[図2]従来の超純水製造装置を示す系統図である。 (符号の説明) * 1 前処理システム

- 2 一次純水システム

3 サブシステム

11 サブタンク

12 ポンプ

13 #0188

1.4 低压紫外線微化装置

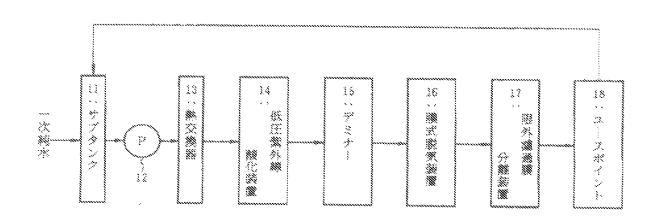
16 イオン交換装置(デミナー)

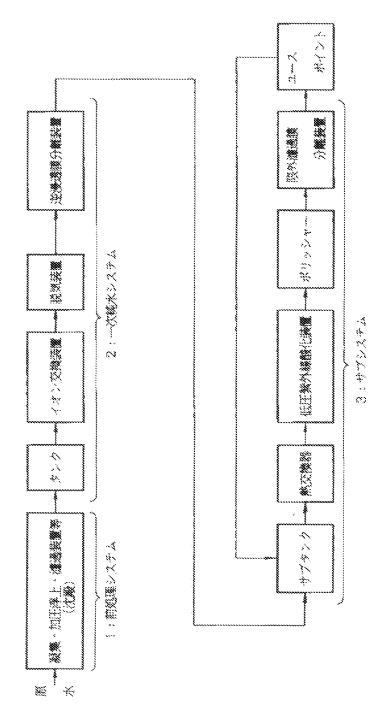
20 10 際式脱氧基础

17 部外液過酸分離装置

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IMI)





PATENT ABSTRACTS OF JAPAN

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(71)Applicant: KURITA WATER IND LTD

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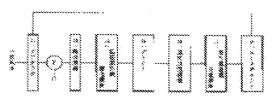
(72)Inventor: FURUKAWA MASAHIRO

KOIZUMI MOTOMU

(54) ULTRAPURE WATER PREPARING APPARATUS

(57)Abstract:

PROBLEM TO BE SOLVED: To efficiently eliminate dissolved oxygen in ultrapure water by arranging a membrane type deaeration device at the rear stage of an ion exchange pure water device in an apparatus wherein the primary pure water is introduced and the water is fed into a subsystem at least with a UV ray irradiation oxidation device and the ion exchange pure water device to obtain an ultrapure water. SOLUTION: In an ultrapure water preparing apparatus for preparing ultrapure water used for cleaning water for semiconductor, the primary pure water obtd. from various pretreatment processes is successively passed through a subtank 11, a pump 12, a heat exchanger 13, a low pressure UV oxidation device 14, an ion



exchange device 15, a membrane type deaeration device 16 and an ultrafiltration membrane separation device 17 and the obtd. ultrapure water with extremely low dissolved oxygen is sent to a use point 18. In the membrane type deaeration device 16, water is made to flow on one side of the membrane and another side is evacuated by means of a vacuum pump to permeate dissolved oxygen through the membrane and to transfer the oxygen on the vacuum side for elimination. It is pref. that the degree of vacuum of this deaeration device 16 is regulated to 55–70Torr and the flow rate of gas such as N2 on the vacuum side is regulated to 5–25% of the amt. of water.

LEGAL STATUS

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[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

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CLAIMS

[Claim(s)]

[Claim 1] Ultrapure water equipments characterized by having arranged the film type deaerator in the latter part of this ion-exchange demineralizer in the ultrapure water equipments which introduce primary pure water, let water flow to the subsystem which has a UV irradiation oxidation system and an ion-exchange demineralizer at least, and obtain ultrapure water.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the ultrapure water equipments with which ultrapure water equipments are started, especially dissolved oxygen concentration obtains very low ultrapure water. [0002]

[Description of the Prior Art] Conventionally, the ultrapure water used as semi-conductor rinse water is manufactured by processing raw water (industrial water, a city water, well water, etc.) with the ultrapure water equipments which consist of a pretreatment system 1, a primary-pure-water system 2, and a subsystem 3 as shown in drawing 2. In drawing 2, the role of each system is as follows.

[0003] In the pretreatment system 1 which consists of condensation, floatation (precipitate), a filter, etc., clearance of the suspended solid in raw water or a colloid substance is performed. In the primary—pure—water system 2 equipped with a reverse osmotic membrane decollator, a deserator, and an ion exchange unit (4 floor 5 column a mixed bed type or type), the ion in raw water and clearance of an organic component are performed. In addition, in a reverse osmotic membrane decollator, TOC of ionicity and colloid nature other than saits clearance is removed. In an ion exchange unit, the ion exchange resin other than saits clearance removes adsorption or the TOC component by which the ion exchange is carried out. Dissolved oxygen is removed in a deserator (nitrogen deseration or vacuum desiring).

[0004] In the subsystem 4 equipped with a heat exchanger, low voltage ultraviolet ray oxidation equipment, a polisher (non-reproducing formula ion-exchange resin equipment), and an ultrafiltration membrane decollator, the purity of water is raised further and it is made ultrapure water, in addition, the 185nm ultraviolet rays taken out with low voltage ultraviolet, ray oxidation equipment, from a low voltage ultraviolet, ray lamp — TOC — an organic-acid pan — GO2 up to — it decomposes. The organic substance and GO2 which were decomposed it is removed by latter ion exchange resin. In an ultrafiltration membrane decollator, a minute particle is removed and the runoff particle of ion exchange resin is also removed.

[Problem(s) to be Solved by the Invention] Although the dissolved oxygen in ultrapure water is a factor important when controlling the thickness of the natural oxidation film of a silicon wafer, dissolved oxygen concentration turns into 5 – 10ppb extent, and it becomes impossible to be satisfied with the case where a still lower dissolved oxygen value is required of demand water quality according to the above-mentioned conventional ultrapure water equipments.

[0006] This invention aims at offering the ultrapure water equipments from which the dissolved oxygen in ultrapure water is efficiently removable.

[0007]

[Means for Solving the Problem] The ultrapure water equipments of this invention are characterized by having arranged the film type deaerator in the latter part of this ion-exchange demineralizer in the ultrapure water equipments which introduce primary pure water, let water flow to the subsystem which has a UV irradiation oxidation system (ultraviolet ray oxidation equipment) and ion-exchange demineralizer at least, and obtain ultrapure water.

[0008] Although dissolved oxygen concentration once fell at the low voltage ultraviolet-ray-oxidation-equipment outlet in a subsystem as a result of this invention persons' investigating transition of the dissolved oxygen concentration in ultrapure water equipments as shown in drawing 2, it went up again at the latter polisher outlet to the dissolved oxygen concentration of a low voltage ultraviolet-ray-oxidation-equipment inlet port (more than it curves depending on the case), and checked that the dissolved oxygen clearance effectiveness of low voltage ultraviolet ray oxidation equipment did not appear as a result. That is, although the dissolved oxygen concentration of the effluent of a primary-pure-water system is about five to 10 ppb and dissolved oxygen concentration is reduced to 0 - 5ppb by processing this water with low voltage ultraviolet ray oxidation equipment, the dissolved oxygen concentration of polisher outlet water is going up to 5 - 10ppb extent again, and the dissolved oxygen concentration of the ultrapure water obtained as a result serves as 5 - 10ppb.

[0009] this invention persons found out that a reaction like following the (1) type had arisen from low voltage ultraviolet ray oxidation equipment between polishers, as a result of examining this phenomenon wholeheartedly. [0010]

[Formula 1]

[0011] That is, although water (H2 O) and TOC exist at a low voltage ultraviolet-ray-exidation-equipment inlet port, when TOC in primary pure water is very low, the amount of UV irradiation in ultraviolet ray exidation equipment becomes superfluous from a design value. (For example, when primary pure water of TOC5ppb flows into the ultraviolet ray exidation equipment which made the amount of UV irradiation the dose corresponding to TOC10ppb, the amount of UV irradiation becomes superfluous by TOC5ppb.) And H2 O is set to H2 O2 (hydrogen peroxide) through OH radical by the exposure of these superfluous ultraviolet rays, and since dissolved exygen is used for TOC decomposition, dissolved exygen concentration falls seemingly. However, H2 O2 generated It is decomposed by the contact catalysis of a polisher (especially anion exchange resin in a polisher), and is O2 again. Since it generates, the dissolved exygen concentration of polisher outlet water goes up.

[0012] It is made for this invention to reduce the dissolved oxygen value in the ultrapure water obtained by removing the dissolved oxygen generated by passing along an ion exchange unit from ultraviolet ray oxidation equipment with a film type deaerator.
[0013]

[Embodiment of the Invention] Hereafter, this invention is explained to a detail with reference to a drawing.
[0014] <u>Drawing 1</u> is the schematic diagram showing one example of the ultrapure water equipments of this invention.

[0015] It lets flow the primary pure water (in the case of usual pure water of 2 or less ppb of TOC concentration) obtained from various head end processes one by one to the subtank 11, a pump 12, a heat exchanger 13, low voltage ultraviolet ray oxidation equipment 14, an ion exchange equipment (DEMINA which mixed an anion exchange resin and cation exchange resin according to the ion load in this example) 15, the film type descrator 16, and the ultrafiltration membrane decollator 17, and the ultrapure water of the obtained super-low dissolved oxygen is sent to the point of use 18.

[0016] What exhaust a sink and another side side for water with a vacuum pump to one deaeration film side, and make the film penetrate, and dissolved oxygen is made to shift to a vacuum side, and was removed as a film type deaerator is used. In addition, since some moisture penetrates the deaeration film and comes out to the vacuum side of this film, it is desirable to remove a sink and moisture for gas, such as nitrogen, and to prevent lowering of membraneous ability to this vacuum side. N2 Regularity is sufficient as a flow rate and it may be fluctuated.
[0017] Although gas, such as oxygen, nitrogen, and a steam, passes the deaeration film, water has a silicone rubber system, a polytetrafluoroethylene system, a polytefine system, a polyurethane system, etc. that what is necessary is just the film which is not penetrated. Various kinds of commercial things can be used as this deaeration film.
[0018] the degree of vacuum of this film type deaerator — 55 – 70Torr and N2 etc. — as for the amount of vacuum side streams of gas, it is desirable to carry out to 5 – 25% of a water flow rate. In addition, although this service condition can be set as arbitration by membraneous ability, its usually above range is desirable. If a degree of vacuum is too low, deaeration effectiveness will fall, if too conversely large, through the film, water will also appear in a transparency side and effectiveness will fall, if too large, a degree of vacuum will not go up but deaeration effectiveness will fall, if too large, a degree of vacuum will not go up but deaeration effectiveness will fall.

[0019] In addition, the configuration of drawing 1 is an example of this invention, and this invention can combine various kinds of devices, as long as it has ultraviolet ray oxidation equipment, the ion exchange unit, and the film type deserator in the subsystem. For example, ultrafiltration (UF) equipment and reverse demotic membrane equipment may be installed after film deseration. Moreover, after carrying out thermal decomposition processing of the raw water under with a pH of 4.5 or less acidity and exidizer existence and decomposing the urea and other TOC components in raw water, the equipment which carries out deionization processing is also incorporable. Ultraviolet ray exidation equipment, an ion exchange unit, and a film type deserator may be installed in multistage. In addition, like drawing 1, by installing UF equipment in the latter part of a film type deserator, the particle generated by the deseration film is removed and the drag-in to the point of use can be prevented.

[0020] In this invention, after contacting the water by which UV irradiation was carried out with ultraviolet ray exidation equipment on ion exchange resin, even if it is important to carry out film deseration processing and it arranges a film type deserator between ultraviolet ray exidation equipment and ion exchange resin, the dissolved exygen in the ultrapure water obtained is not reduced.

[Example] The example of a comparison and an example are given to below, and this invention is more concretely explained to it.

[0022] Example of comparison 1 raw water (Atsugi city water TOC700 - 800ppb, dissolved oxygen 8000ppb, electric conductivity S/cm of 240micro) was processed with the equipment of <u>drawing 2</u>, and ultrapure water was manufactured.

[0023] Made one to example 3 subsystem into the thing of <u>drawing 1</u> incorporating a film type deaerator, and the service condition and the amount of water flow of a film type deaerator were carried out as a table 1, and also otherwise water was manufactured like the example 1 of a comparison.

[0024] The specification of each equipment in this example of a comparison and example is as follows.

[9025] Low-voltage UV-irradiation oxidation system: 80Wx4 ** (0.32kW)

(Generating wavelength: 185nm and 254nm)

polisher (DEMINA): — mixed bed type ion exchange unit SV=70 which carried out mixed filling of cation—exchange—resin and anion exchange resin = 80hr=1 ultrafiltration—membrane decollator: — KU-1010 (Kurita Water Industries, Ltd. make)

deaeration film: Hoechst Celanese Make Liqui-Cel 1 housing: --- 5PCH-120 cartridge: --- 5PCH-118 and SN: --- 1221 [0026]

[A table 1]

	水流量	接式股気装置			
	(of/fir)	真空度 (Terr)	N's 流量 (Na*/H)		
実施例1	1.8	70	0.5		
実施例 2	4	5 5	0.8		
突施例3	3	65	0.5		
計数例 1	3				

[0027] The dissolved oxygen of the electric conductivity of the obtained ultrapure water and the effluent of each equipment after low voltage ultraviolet ray oxidation equipment was as being shown in a table 2. [0028]

[A table 2]

	南 存 酸 素 (ppb)					fersh s.	
	一次純水	紫外線酸化 装置出口	が出口	脱领装置 出口	urwo	程導度 (μS/om)	
実施例 1	< 5	<5	9	1	1.	18.24	
実施例2	< 5	< 5	7	< 1	< 1	18.24	
实施例3	< 5	< 8	7	< 1	<1	18, 24	
1640911	<.5	< 5	7~10		7~11	18.24	

[0029] According to the example of this invention, it is clearer than a table 2 that ultrapure water with very low dissolved oxygen concentration is obtained. [0030]

[Effect of the Invention] According to the ultrapure water equipments of this invention, dissolved oxygen concentration can manufacture remarkable low ultrapure water as explained in full detail above.

Drawing selection drawing 1

1000

